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CVD OF AlN AND SiC USING CYCLIC ORGANOMETALLIC PRECURSORS

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ABSTRACT

The use of cyclic organometallic molecules as single-source MOCVD precursors is illustrated by means of examples taken from our recent work on AlN and SiC deposition, with particular focus on SiC. Molecules containing (AlN)₃ and (SiC)₂ rings as the "core structure" were employed as the source materials for these studies. The organoaluminum amide, [Me₂AlNH₂]₃, was used as the AlN source and has been studied in a molecular beam sampling apparatus in order to determine the gas phase species present in a hot-wall CVD reactor environment. In the case of SiC CVD, a series of disilacyclobutanes, [Si(XX')CH₂]₂ (with X and X' = H, CH₃, and CH₂SiH₂CH₃), were examined in a cold-wall, hot-stage CVD reactor in order to compare their relative reactivities and prospective utility as single-source CVD precursors. The parent compound, disilacyclobutane, [SiH₂CH₂]₂, was found to exhibit the lowest deposition temperature (ca. 670 °C) and to yield the highest purity SiC films. This precursor gave a highly textured, polycrystalline film on the Si(100) substrates (70% with a SiC<111> orientation).

INTRODUCTION

The use of single-source precursors for MOCVD of refractory materials such as AlN and SiC affords potential advantages in terms of controlling the composition, deposition temperature and microstructure of the deposited product. The need to control these factors may be particularly critical in a CVI reactor environment, where the thermal/chemical stability of the fiber reinforcement, or of the fiber-matrix interface, may set limits on the processing temperature or where the use of mixed precursor sources may lead to local variations in composition and/or microstructure [1].

To date, the choice of organometallic molecules for use as single-source precursors has been largely an empirical process, with few guidelines available relating to the relationship between molecular structure and such factors as decomposition temperature, product composition, and microstructure. In the case of SiC CVD, precursors such as methyltrichlorosilane (MTS) and the methylsilanes have been employed for many years; however, temperatures well in excess of 1000 °C along with added H₂ are usually needed to obtain SiC free of elemental C or Si and significant variations in composition are often experienced as the deposition conditions are varied [1,2]. Moreover, the HCl produced as a byproduct of the decomposition of chlorosilanes can be corrosive to fiber

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reinforcements or Si substrates [1].

The Use of Cyclic Organometallics as Precursors to AlN and SiC

A key objective of our research efforts has been to develop precursors and procedures that will be effective in depositing refractory materials such as AlN and SiC in high purity at relatively low temperatures ($< 500^{\circ}\text{C}$) and pressures (< 10 torr) under thermal CVD conditions. Such procedures would be advantageous for many applications for CVD in electronics or in the processing of structural components where the substrate, the interface chemistry or the device structures built into the substrate may set effective limits on the processing temperature.

Another major objective of our program has been to obtain a detailed understanding of selected thermal CVD processes and, through this understanding, to develop precursor structure/function relationships that will be useful in the design of new precursor systems. In this context, we have selected two particular systems for detailed study, leading to the production of AlN and SiC thin films, respectively. In both cases the selected precursor systems are cyclic compounds which provide all of the required elements of the product thin film in the form of a volatile molecular entity of the type, $[\text{A}(\text{XX}')\text{B}(\text{YY}')]_n$ (where A, B = Al, N; or Si, C; X, X', Y, Y' = H, CH_3 , $\text{CH}_2\text{SiH}_2\text{CH}_3$ etc. and $n = 2$ and 3). Our prior studies have provided ample evidence for the efficacy of such cyclic species as both AlN and SiC precursors, as well as a base of fundamental information relating to their synthesis, properties and pyrolysis chemistry [3]. As a class of molecular species, such cyclic compounds often afford higher volatilities compared to comparable acyclic compounds, perhaps due to their more compact molecular structures, as well as a core structural unit that can be used as a framework for attaching different substituent groups.

AlN Deposition Using Organoaluminum Amides

In the case of the cyclic organoaluminum amides of the type, $[\text{R}_2\text{AlNR}'_2]_n$, both dimeric ($n = 2$) and trimeric ($n = 3$) structures are known, depending on the nature of the R and R' groups; however, the most volatile species are those with relatively simple (R, R' = Me or H) substituents [4]. The compound $[\text{Et}_2\text{AlN}_3]_3$ has been studied by Gladfelter and coworkers and is reported to give films at quite low deposition temperatures; however, C-contamination appears to be a significant problem [5]. We have examined a series of N-Me and N-H amides and have found that, of these, only $[\text{Me}_2\text{AlNH}_2]_3$ gives satisfactory results as a MOCVD precursor for AlN [6].

Our initial studies of this precursor were carried out in a simple hot-wall CVD reactor and resulted in the deposition of high quality, polycrystalline thin films of AlN on Si and other substrates at considerably lower temperatures than had been required previously to obtain films of this type [7]. The apparent resemblance between the six-membered



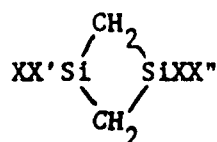
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(AlN)₃ rings in this precursor and those that make up the wurzite structure of AlN provided an additional stimulus for a more detailed study of the precursor-to-ceramic conversion process in this case. However, separate studies of this precursor system in solution, by NMR spectroscopy, revealed a rapid equilibrium involving the dimeric and, presumably, the monomeric form of this compound [8] and Generalized Valence Bond calculations were performed that indicated a planar structure and a significant stability for this monomer [9]. Recent mass spectral studies carried out in collaboration with J. Hudson have suggested that this trimer-dimer equilibrium extends also to the gas phase and that the active species in the CVD process may be one of these other forms of the [Me₂AlNH₂]₃ precursor [10]. We are currently engaged in a detailed study of the gas phase chemistry of the [Me₂AlNH₂]₃ compound by using a molecular beam mass spectrometry system to sample the gas phase species from a reactor held at different temperatures and pressures. Time-of-flight measurements are being carried out in this system in order to distinguish the parent molecular species from fragments produced in the mass spectrometer. The objective of these studies is to gain a fuller understanding of the gas phase chemistry occurring in a hot-wall CVD reactor using the [Me₂AlNH₂]₃ compound.

SiC Deposition with Disilacyclobutanes

Our study of SiC precursors has centered on a series of disilacyclobutanes which contain the (SiC)₂ ring but which differ in the nature of the substituents that are attached to this ring. The objective here is to examine a homologous series of compounds with a common structural core, so as to determine how the nature of the substituent influences the decomposition chemistry and nature of the product film. The choice of the disilacyclobutanes as the core structure for this study was motivated by the results of prior decomposition studies of organosilicon compounds and the supposition of significant ring strain energy stored in the relatively small four-membered (SiC)₂ ring [11]. In comparison with the organoaluminum amides studied as AlN precursors, it should be noted that the nature of the bonding and the chemistry in these (SiC)₂ compounds differs considerably, with a greater prevalence of covalent bonding and radical decomposition processes in the case of the organosilicon species.

The specific compounds chosen for study in this initial examination of substituted disilacyclobutanes as SiC precursors are members of the structural type,



where: X = Me, X' = H, X'' = CH₂SiH₂CH₃ (I)

X = Me, X', X'' = H (II)

and X, X', X'' = H (III)

Preliminary results obtained for the initial compound in this series, $[\text{MeSi}(\text{H})\mu(\text{CH}_2)_2\text{Si}(\text{Me})\text{CH}_2\text{SiH}_2\text{Me}]$ (I), along with its synthesis, were described in earlier publications [12]. We have now prepared the dimethyldisilacyclobutane (II), as well as the parent disilacyclobutane, $[\text{SiH}_2\text{CH}_2]_2$ (III), and obtained information regarding their decomposition chemistry and relative utility as SiC precursors. The results of these studies are reported briefly herein.

EXPERIMENTAL

Preparation of Precursors

1,2-Dimethyldisilacyclobutane was prepared as the 50/50 *cis/trans* mixture by using a modification of the procedure described by Kriner [13]. This involved the Grignard coupling of $\text{CH}_3\text{Cl}(\text{OEt})\text{SiCH}_2\text{Cl}$ with Mg in THF, followed by reduction with LiAlH_4 in THF. The resultant mixture, consisting of mainly the four- and six-membered $[\text{Si}(\text{Me})\text{HCH}_2]_n$ ring compounds, was purified by atmospheric pressure fractional distillation (under N_2) giving an overall yield of ca. 25% for the disilacyclobutane product mixture (b.p. 96-98 °C).

Disilacyclobutane was also prepared by using a method described in the literature [14], which involved the pyrolysis of 1,1-dichloro-1-silacyclobutane vapor by passing it through a 840 °C furnace tube packed with porcelain saddles. The resultant 1,1,3,3-tetra-chloro-1,3-disilacyclobutane was purified by sublimation and reduced in $n\text{-Bu}_2\text{O}$ with LiAlH_4 . The overall yield was on the order of 30%.

Both compounds were characterized by ^1H , ^{13}C , and ^{29}Si NMR spectroscopy, as well as by mass spectrometry, and were >95% pure.

CVD Apparatus

A home-built, cold-wall, hot-stage CVD apparatus was used for these studies that was described briefly in a previous paper [12b]. The apparatus includes a stainless steel reactor chamber which contains the heated substrates, a bubbler loaded with the precursor, and a vacuum system consisting of a turbomolecular pump backed by a two-stage, rotary vane mechanical pump. The Si(100) substrates (resistivity = 1.1 Ω cm.; phosphorous doped) were cleaved into ca. 1.4 cm X 6.5 cm rectangular sections. Two or three of these substrates were then connected in a series circuit using tungsten clips with copper leads and resistively heated by using a current-limited DC power supply. The temperature is measured by means of a thermocouple attached to one of the wafers. The thermocouple was calibrated throughout the temperature range of interest prior to deposition under 1 torr Ar pressure by using an optical pyrometer. The operating pressure in the reactor is monitored by a temperature-controlled

capacitance monometer and controlled during the deposition by means of an electronically operated butterfly valve between the pumping system and the reactor chamber.

Decomposition Onset Determination

The CVD experiments using the LPCVD system described above were all carried out using a mass flow controlled argon carrier gas (10.0 sccm) while maintaining a constant reactor pressure of 1.0 ± 0.1 torr. The precursor was loaded into a stainless steel bubbler while inside a N_2 -dry box. Once the bubbler was attached to the CVD system, the precursor was subsequently freeze/thaw-degassed (backfilling with argon to remove the $N_2(g)$) before the start of the CVD experiment. During each LPCVD experiment, the partial pressure of the precursor was maintained at ca. 2.0 torr by cooling the bubbler with a slush bath as needed.

For the decomposition onset determination, a quadupole mass spectrometer was connected to the reaction chamber just downstream of the heated wafer sections. A series of liquid N_2 -cooled traps were placed in the line leading to the mass spectrometer in order to trap out unreacted precursor and condensable byproducts. While flowing the precursor continuously through the reactor, the temperature of the Si(100) substrates was increased incrementally, as the intensity of the $m/e = 15, 16$ peaks for methane and $m/e = 2$ for hydrogen were monitored in the mass spectrometer. After each temperature increase, a reading was taken at each m/e setting until a relatively constant value was obtained. After reaching a maximum of ca. $900^\circ C$, the substrate temperature was then decreased in intervals, providing measurements in both directions. During the long period over which these measurements were performed (several hours), an appreciable thickness of SiC was built up, insuring a fresh "SiC" surface throughout these decomposition studies.

RESULTS AND DISCUSSION

In our initial studies of SiC MOCVD we employed a substituted disilacyclobutane, 1,3-dimethyl-3-methylsilamethylene-1,3-disilacyclobutane $[MeSi(H)\mu(CH_2)_2Si(Me)CH_2SiH_2Me]$ (I), that was obtained as the major volatile product from the Grignard coupling of $MeSiCl_2CH_2Cl$, after reduction [12,13]. Its ease of synthesis, compared to the simpler unsubstituted and methyl-substituted derivatives, and the observation that it gave near stoichiometric SiC films in a hot-wall CVD reactor environment, makes it an attractive choice as a CVD precursor. However, subsequent studies in our cold-wall reactor indicated that the composition of the SiC product varied considerably with the deposition conditions employed and that temperatures in excess of $750^\circ C$ were needed to effect decomposition.

The present study set out to compare this precursor with two related

disilacyclobutanes, 1,2-dimethyldisilacyclobutane $[\text{Si}(\text{Me})\text{HCH}_2]_2$ (II), and disilacyclobutane itself, $[\text{SiH}_2\text{CH}_2]_2$ (III). These precursors differ in the number of H's attached to the Si atoms of the disilacyclobutane ring, with 1, 2, and 4 H's present in (I), (II) and (III) respectively. They also differ in volatility, with the vapor pressure at 25 °C increasing from ca. 2 to 46 and 305 torr from (I) to (III).

(insert Figure 1. Structures of disilacyclobutanes used in this study)

The results of the decomposition onset study for compound (I) are shown in Figure 2, and indicate a decomposition onset for this precursor of approximately 760 °C. Similar measurements were performed on both compounds (II) and (III), indicating decomposition onset temperatures of 740 and 670 °C, respectively. It is notable, however, that in the case of compound (III), no appreciable methane byproduct was observed, thus only the hydrogen m/e = 2 peak was employed to determine the decomposition temperature for this precursor.

(insert Figure 2. Deposition onset determination for compound I)

Separate studies in which the substrates were held at the above onset temperatures but where the temperature of the substrates varied locally indicated that these decomposition onset temperatures also correspond approximately to the minimum temperatures for the production of a deposit.

The next series of experiments involved deposition for an extended period (ca. 30 min) at a particular temperature (ca. 700, 800, 900, 1000 and 1100 °C) so as to deposit at least 2 μm of the product film. The substrates were then removed from the reactor for compositional analysis by Auger spectroscopy. The deposition rates in these experiments varied from about 0.5 to 15 μm/h and the pressure in the reactor, as in the deposition onset studies, was maintained at 1 μm total pressure.

The results show that the films obtained from precursors (I) and (II) under these conditions contain excess carbon, (as much as 16 atm % for precursor (I) at 760 °C) but that these films approach the stoichiometric SiC composition as the substrate temperature reaches around 1000 °C (Figure 3). On the other hand, precursor (III) appears to give Si-rich films (ca. 6%) at the lowest temperature studied (700 °C), but reaches the stoichiometric ratio by 800 °C.

(Figure 3. SiC composition as a function of temperature for films deposited by using compound I)

Powder X-ray diffraction studies were also carried out on these films using a pole figure diffractometer to analyze for possible preferred orientation. XRD patterns consistent with β-SiC were observed for films deposited by using all three precursors; however, in the case of precursors (I) and (II), only the films obtained at the higher temperatures (>900 °C) exhibited significant crystallinity. For precursor (III) on the other hand, even at the lowest deposition temperature studied (800 °C) the

films were clearly crystalline. In this case, a pole figure analysis was performed on the film deposited at 945 °C, which indicated a strong fiber SiC(111) texture.

Thus, for these three precursors, the onset of crystallinity correlates with the composition of the product films and, as the composition approaches 1:1, increased crystallinity is evidenced. This is consistent with expectations regarding the inability of the crystalline SiC phases to tolerate significant variations from the 1:1 stoichiometry and the prior observations that excess C (or Si) tends to segregate to the SiC grain boundaries, thereby inhibiting crystallization [15]. The observation of preferred orientation of the SiC crystallites in the film obtained from precursor (III) at 945 °C can be also be understood on the basis of prior work, in which it was concluded that the SiC(111) plane would be preferred for crystal growth because it has a surface energy minimum resulting from a high atomic packing density [16].

SEM and FTIR transmission studies were also carried out on the SiC-coated Si wafer sections. The SEM studies indicated a wide variation of surface and cross-sectional film morphologies ranging from apparently fine grained, dense structures to rather porous deposits with elongated grains depending on the precursor employed and the deposition temperature. In general, the films were well adhered to the Si surface and had a rough, grainy, surface morphology. The FTIR studies showed only the expected 800 cm⁻¹ band characteristic of SiC with no absorption bands in either the C-H or Si-H stretching regions.

After completion of a series of depositions at different temperatures for each precursor, the byproducts that had condensed in the main liquid N₂ cold trap in the CVD apparatus were analyzed by using both G.C./FTIR and mass spectrometry. In the case of (I), a mixture of hydrocarbons and methylsilanes were observed, including ethane, ethylene, acetylene, plus trimethyl- and tetramethylsilane. In the case of (II), the same C₂ hydrocarbons, along with methyl-, dimethyl- and trimethylsilane were observed, but no tetramethylsilane. Both precursors also gave hydrogen and methane as byproducts, as indicated from the decomposition onset studies.

In contrast, the byproduct distribution obtained from precursor (III) was relatively sparse, with only traces of the C₂ hydrocarbons along with methyl- and dimethylsilane detected, in addition to the hydrogen noted in the deposition onset determination.

Both the gas products observed and the compositional variations in the product films obtained using these three precursors can be understood on the basis of prior mechanistic studies of decomposition reactions of these and related disilacyclobutanes. Disilacyclobutane (III) and dimethyldisilacyclobutane (II), along with the tetramethyl compound, have been studied in a stirred flow reactor by Auner and coworkers [11b]. The results of these and other studies [11a,14,17] have suggested that the decomposition onset temperature for these disilacyclobutanes varies inversely with the number of H-atoms substituted onto the Si atoms of the rings, with the parent disilacyclobutane decomposing at the lowest temperature. This presumably relates to the tendency of these

H-substituted disilacylobutanes to form reactive silylenes (:SiRR' species) by 1,1- H_2 elimination or 1,2 Si-to-C H-transfer reactions that again form a silylene with resultant ring opening.

The product distribution from the stirred flow reactor study also parallels that observed herein for these precursors, where the distribution of methylsilanes observed reflects the bonding of Si in the initial precursor. Thus only compound I, which has up to four carbons around one Si, gives any observed tetramethylsilane, compound II gives trimethylsilane as the highest methylated silane, and III, disilacyclobutane, gives only up to dimethylsilane. This suggests that molecular rearrangements involving H transfer, as opposed to radical processes, are probably dominant in determining the silane reaction byproducts. Clearly much more needs to be done on these systems before a detailed reaction mechanism can be discussed with any confidence. Further deposition experiments along with surface decomposition studies are planned in order to help answer these remaining questions.

In terms of comparing the three precursors as SiC CVD precursors, clearly the parent disilacyclobutane comes out as the preferred compound for use in situations where the product purity and/or deposition temperature are critical factors. On the other hand, the preparation of this compound is still relatively difficult and it is certainly too costly to use at present for large scale CVD or CVI applications. Efforts to obtain this compound by more convenient methods and to examine alternative precursors of this type are also in progress.

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